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Part translation

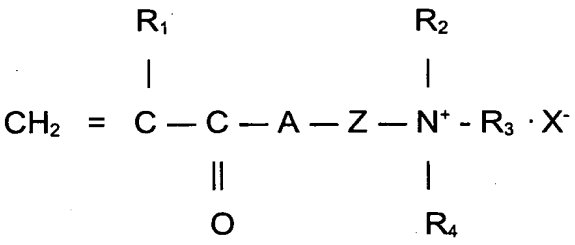
(Laid open print)

Citation 2:

Japanese Patent Laid Open Print No. 06-157668 of 07.06.1994
Application No. 04-339502 of 27.11.1992
Applicant: Showa Denk K. K., Tokyo, Japan
Title: **Water-in-Oil Cationic Emulsion, its Preparation and its Use as Polymeric Flocculant**

Claims:

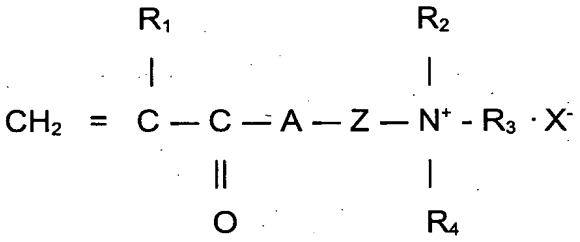
1. Water-in-oil cationic emulsion whereby a cationic vinyl monomer of the general formula



wherein
A is an oxygen atom or NH,
Z is C_{1-4} alkylene or C_{2-4} hydroxyalkylene,
R_1 is hydrogen or methyl,
R_2 is C_{1-14} alkyl, C_{2-4} hydroxalkyl or benzyl,
R_3 and R_4 are independently C_{1-4} alkyl or C_{2-4} hydroxalkyl and
X is a salt-forming anion,
homopolymerizes a hydrophobic organic liquid and a nonionic surfactant in the presence of water and/or copolymerizes with other water-soluble vinyl monomers,
characterised in that

(A) the concentration of the polymers is 20 to 70 weight %,
(B) the concentration of the hydrophobic organic liquid is 10 to 50 weight %,
(C) the concentration of the nonionic surfactant is 8 weight % or less, and
(D) the weight-mean particle diameter of the polymer emulsion is 0.3 µm or less.

2. A method for the manufacture of a water-in-oil cationic emulsion whereby a cationic vinyl monomer of the general formula



wherein
A is an oxygen atom or NH,
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R_2 is C_{1-14} alkyl, C_{2-4} hydroxalkyl or benzyl,
R_3 and R_4 are independently C_{1-4} alkyl or C_{2-4} hydroxalkyl and
X is a salt-forming anion,
homopolymerizes a hydrophobic organic liquid and a nonionic surfactant in the presence of water and/or copolymerizes with other water-soluble vinyl monomers,
characterised in that
prior to the start of the polymerization, a stable water-in-oil monomer emulsion, wherein the weight-mean particle diameter of the polymer emulsion particles is 0.3 µm or less, is formed.

3. Polymer flocculant consisting essentially of a water-in-oil polymer emulsion according to claim 1.

Detailed description of the invention (excerpt):

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[0018]
It is important for the present invention that a stable water-in-oil monomer emulsion with a weight-mean particle diameter of 0.3 µm is formed prior to the polymerisation. In general, a polymer emulsion can be obtained with uniformly fine particles in which a fine monomer emulsion is formed prior to the polymerisation. In this case, after the polymerisation, the polymer emulsion has a high molecular weight, storage stability and increased water solubility. When the weight-mean particle diameter of the monomer emulsion exceeds 0.3 µm it is difficult to achieve a polymer emulsion with a high molecular weight and good storage stability.

[0019]
The maintenance of a monomer emulsion with a weight-mean particle diameter of 0.3 µm or less requires sufficient monitoring of the composition proportions between monomers, water, the hydrophobic organic liquid and the nonionic surfactant, as well as of the types of hydrophobic organic liquid and nonionic surfactant. This mixture also needs to have a very strong shear stress. Homoginising apparatus, in-line homomers, etc can be used to impart shear stress, whereby it is important to keep a sufficiently high average circulation count (the average thereof, that the liquid flows between the blades and the outer walls).

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[0025]
[Example]
The present invention is further explained by, but is not limited to, the examples.

[Example 1]
640 g of n-hexane as hydrophobic organic liquid was mixed with 80 g of polyoxyethylenestearylether as nonionic surfactant in a beaker with a volume of 2 litres in order to produce an oil solution. 316 g of ion exchange water with 360 g (80% aqueous solution) of dimethylaminoethylmethacrylate hydrochloride and 192 g of acrylamide dissolved therein was placed in a further beaker with a volume of 1 litre in order to produce an aqueous monomer

solution. The aqueous monomer solution was slowly added to the oil solution while stirring with an homomer apparatus at 8 000 rpm. Following completion of the addition, the emulsification dispersion was stirred for a further 180 minutes (average circulation count: 3 000) in order to produce a monomer emulsion. The entire monomer emulsion was poured into a 2 litre three-necked flask with a stirrer, a thermometer and a nitrogen inlet, and nitrogen gas with a flow rate of 1.5 litres/minute was introduced for 120 minutes in order to degas the monomer emulsion. Finally, the temperature of the monomer emulsion was kept at a constant 35 °C and 28 g of a 0.1 % aqueous ferrous chloride solution and 14 g of 0.1 % ammonium persulfate were then added as polymerisation initiators in order to begin the polymerisation. The polymerisation was completed after 4 hours, whereby a water-in oil cationic polymer emulsion with a weight-mean particle diameter of 0.21 µm (measured by the light diffusion process) was obtained.

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